

LIST OF U.S. CUSTOMS LABORATORY METHODS

USCL NUMBER	METHOD	TITLE
71-01	USCL Manual	<u>Gold - Quantitative Analysis by Fire Assay Method</u>
71-02	ASTM E 56	<u>Test Method in Standard Test Methods for Chemical Analysis of Silver Brazing Alloys</u>
71-03	ASTM E 46	<u>Test Methods for Chemical Analysis of Lead- and Tin-Base Solder</u>
71-04	USCL Manual	<u>Gold Fineness in Articles of Jewelry</u>

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 71-01

INDEX

Gold: Quantitative Analysis by Fire Assay

certain Customs' samples.

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

0 INTRODUCTION

The quantification of gold is needed to ensure compliance with the Gold Labeling Act of 1976 (Public Law 94-450) which is based on criteria set forth in U.S. Code Title 15 - Commerce and Trade parts 294 and 295. Also, depending on the product classification in the HTSUS may be contingent upon the gold content; statistical reporting for tariff purposes is also based on gold content.

1 SCOPE

1.1 The procedure discussed in this method is a modification of the "Fire Assay Method for Gold Analysis". Minor modifications were made to the method, as provided below, to accommodate

2 REFERENCES

The Fire Assay For Gold And Silver,
Scott's Standard Methods of Chemical Analysis, Volume I, 5th edition
N.H. Furman, editor
Van Nostrand, New York, 1939

ASTM E 1335
Standard Test Method for Determination of Gold in Bullion by Cupellation

3 REAGENTS AND APPARATUS

- 3.1 Parting acid I (2:3) A.C.S. reagent grade nitric acid
- 3.2 Parting Acid II (2:1) A.C.S. reagent grade nitric acid
- 3.2 Silver powder 99.99% (60 mesh)
- 3.3 Pure bone cupels (1.5 inch)
- 3.4 Assay Grade pure lead foil 76 mm X 0.15 mm
- 3.5 50 mL Erlenmeyer flasks

- 3.6 30 ml porcelain crucibles
- 3.7 CEM crucibles (disposal)
- 3.8 Smooth jawed needle-nosed pliers
- 3.9 Stainless hammer and anvil (4" X 4")
- 3.10 Stainless forceps
- 3.11 Analytical balance (± 0.0001 g)
- 3.12 Muffle furnace (+1000EC)
- 3.13 Pellet press (20 - 25 ton capacity) and 1.25 inch pellet die
- 3.14 Steam bath

4 PREPARATION OF SAMPLE

- 4.1 Cut the sample into small pieces (note whether the pieces are cut from soldered or unsoldered areas).
- 4.2 Clean and degrease the cut pieces with methanol.
- 4.3 Dry in an oven at 100EC for one (1) hour and cool in a desiccator for 30 minutes.

5 PROCEDURE

- 5.1 Cut a piece of the pure assay-grade lead foil into a size/weight that corresponds to the sample weight in a ratio of 2.4 grams of foil to 0.1 grams of sample. A

cork borer sharpener can be used as a form to shape the pieces of foil into a cone.

- 5.2 Weigh enough of the cleaned and dried sample into the cone to give a final weight of approximately 120 milligrams of pure gold.
- 5.3 The silver powder (inquart) is added to the cone in a ratio of 2.5 parts of silver to 1 part sample, by weight.
- 5.4 The cone is sealed with the smooth jawed needle nosed pliers and placed in a preheated pure bone cupel at 1000 - 1100EC in the muffle furnace.
- 5.5 The sample is heated 25 - 35 minutes until a shiny well formed bead (dore' bead) is obtained.
- 5.6 After cooling, the bead is removed from the cupel with the stainless steel forceps, cupel fines are removed with a stiff brush, and hammered (approximately 12 strokes) on the stainless steel anvil.
- 5.7 The flattened bead is annealed in a CEM crucible at 700EC for 4 minutes.
- 5.8 The cooled bead is pressed at 20 - 25 thousand pounds in the pellet press so that the bead is flattened to approximately 0.05 mm thick.
- 5.9 Repeat step 5.7.

- 5.10 The flattened bead is cooled and placed in a 50 mL Erlenmeyer flask with 20 mL of hot (90 - 100 EC) parting acid I. The sample is heated at 90 - 100 EC for 30 minutes, the acid decanted, and the sample washed with hot distilled water.
- 5.11 20 mL of hot (90 -100EC) parting acid II is added and the sample is again heated and then washed as in step 5.10.
- 5.12 The flattened bead is transferred to a tared (previously heated to 700EC) 30 mL porcelain crucible, the liquid is evaporated off on a steam bath, and then placed in the muffle furnace at 700EC for 2 minutes. The crucible is cooled in a desiccator for at least 60 minutes and weighed on an analytical balance.

6 CALCULATIONS

- 6.1 The karat gold is calculated as:

$$\text{karat gold} = 24 \text{ karats} \times$$

$$\frac{(\text{wt of crucible} + \text{sample} - \text{wt of crucible})}{\text{weight of sample taken}}$$

- 6.2 The percent gold is calculated as:

$$\text{percent gold} = 100 \times$$

$$\frac{(\text{wt of crucible} + \text{sample} - \text{wt of crucible})}{\text{weight of sample taken}}$$

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 71-02

Index

ASTM E 56 Test Method in Standard Test Methods for Chemical Analysis of Silver Brazing Alloys

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This method covers the chemical analysis of silver brazing alloys. This is one of the methods that can be used to determine the composition of silver and articles thereof in Chapter 71 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM E 56

Test Method in Standard Test Methods for Chemical Analysis of Silver Brazing Alloys

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 71-03

Index

ASTM E 46 Test Methods for Chemical Analysis of Lead- and Tin-Base Solder

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

These methods cover procedures for the chemical analysis of lead and tin-base solder. These are among the methods that can be used to determine the composition of lead and articles thereof provided for in Chapter 71 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM E 46

Test Methods for Chemical Analysis of
Lead- and Tin-Base Solder

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 71-04

INDEX

Gold Fineness in Articles of Jewelry

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

These procedures may be used in the analysis of articles of gold used for personal adornment for value determination for tariff purposes. These articles (covered under the Harmonized System Tariff Numbers 7113.19.10-7113.19.50, 7114.19.00 and 7115.90.10) include small objects of personal adornment (gem-set or not) such as rings, bracelets, necklaces, brooches, ear-rings, neck chains, watch-chains, and other ornamental chains; fobs, pendants, tie-pins and clips, cuff-links, dress studs, buttons, etc; religious or other

crosses; medals and insignia; hat ornaments (pins, buckles, rings etc.); ornaments for handbags; buckles and slides for belts, shoes, etc; hair-slides, tiaras, dress combs and similar hair ornaments.

If the gold content of the article is required to determine if it meets the standard of fineness (the gold content expressed as parts per thousand) required by the Gold Labeling Act of 1976 (public Law 94-450), the recommended method is USCL method 71-01, Gold - Quantitative Analysis by Fire Assay.

2 REFERENCES

Gold Labeling Act of 1976
Public Law 94-450

"Precipitation with Sulfurous Acid"

Standard Methods of Chemical Analysis

N.H. Furman, Editor, Volume 1
D. Van Nostrand Co., Inc., 1962,
pg 472.

3 PRINCIPLE

The two procedures which are described may be used in the analysis of articles of gold used for personal adornment. The procedure of choice depends upon the questions to be answered. First is a non-destructive screening procedure using X-Ray fluorescence. The second is a gravimetric determination.

4 APPARATUS, REAGENTS AND MATERIALS

- 4.1 Energy dispersive X-ray spectrometer system with a fundamental parameters program for XRF analysis
- 4.2 Hydrochloric acid, reagent grade
 - 4.2.1 Hydrochloric acid, 1% (v/v) solution
- 4.4 Nitric acid, reagent grade
- 4.5 Aqua regia: 4 parts HCL, 1 part HNO₃
- 4.6 Sulfurous acid, saturated, reagent grade
- 4.7 Filter paper, close-grained Whatman N. 44
- 4.8 Standard laboratory equipment: including hot plates, steam bath, drying oven, filter funnels,

Bunsen burners, graduated cylinders, litmus paper, beakers, Erlenmeyer flasks

- 4.9 Analytical balance, ± 0.0001 gram

5 SAMPLE PREPARATION

- 5.1 Analysis by XRF:
 - 5.1.1 Clean the surface of any dust or dirt. Remove any organic coating.
- 5.2 Analysis by sulfurous acid precipitation:
 - 5.2.1 First clean the surface of any dust or dirt.
 - 5.2.2 Cut a portion of the sample off using cutters or other appropriate means. This portion should weigh approximately 1-2 grams.
 - 5.2.3 Weigh each portion to ± 0.0001 grams.

6 PROCEDURE FOR ANALYSIS BY XRF

- 6.1 Set up XRF instrument as directed by manufacturer's directions. The following are general guidelines:
 - a) X-ray current is adjusted to give approximately 50% dead time.
 - b) As a secondary target use gadolinium or other suitable target.

- c) Acquisition time is 100 seconds after correction for dead time. 100 mL .
- 6.2** Follow manufacturer's directions for operation of instrument and fundamental parameters program for XRF analysis.
- 6.3** Follow manufacturer's directions for calibration.
- 6.4** Choose a portion of the sample with a flat surface and the greatest area. The surface presented to the X-ray beam should be homogeneous and not coated with organic material.
- 7 PROCEDURE FOR PRECIPITATION WITH SULFUROUS ACID**
- 7.1** Dissolve the sample in aqua regia.
- 7.2** After solution of the alloy or residue in aqua regia, any insoluble matter is filtered from the solution.
- 7.3** Carefully evaporate the solution on a steam bath.
- 7.4** Two additions, each of 5 mL of hydrochloric acid may be necessary to decomposed and volatilize the nitrates.
- 7.5** Dilute the solution to contain not more than 100 mg of gold per
- 7.6** Let the solution stand for several hours. (See note **8.2**)
- 7.7** Remove any insoluble matter by filtration.
- 7.8** Heat the clear solution to 80-90EC.
- 7.9** Slowly add 20 mL of a saturated solution of sulfurous acid.
- 7.10** Heat the solution gently until the gold precipitate coagulates.
- 7.11** Add an additional 5 mL of the sulfurous acid solution and continue the heating for 10 minutes to insure complete precipitation of gold.
- 7.12** Filter the cooled solution a close-grained filter paper.
- 7.13** Wash the paper thoroughly with warm 1% hydrochloric acid and then to neutrality with water.
- 7.14** Dry the paper and ignite.
- 7.15** After cooling, weigh the precipitate of gold.
- 8 NOTES ON THE PROCEDURE**
- 8.1** Previous experience with the XRF method showed deviations of approximately +/- 1 karat or %.

- 8.2** Care must be taken in filtering gold chloride not to expose the solution in contact with the filter paper to sunlight since gold is photosensitive and under such conditions will be reduced in the filter paper.